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Indian Standard

CHEMICAL ANALYSIS OF FERROTITANIUM

PART 5 DETERMINATION OF SULPHUR BY DIRECT COMBUSTION METHOD

ICS 77.100 ; 77.120.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

FOREWORD

This Indian Standard (Part 5) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' covers the chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard the Sectional Committee decided that a separate series for each ferroalloy be prepared. Accordingly, IS 1559 in its various parts was published for the methods of chemical analysis of ferrosilicon.

The methods of chemical analysis of various constituents of ferromolybdenum and ferrochromium are covered in a series of parts of IS 12614 and IS 13452 respectively. The chemical analysis of ferrotitanium is covered in a series of parts of this standard. With the publication of these parts the analysis of relevant constituent prescribed for ferrotitanium in IS 1559 : 1967 will be superseded. This part covers the method for determination of sulphur by direct combustion method. The other parts of this series are:

Part 1	Determination of carbon by the direct combustion (gravimetric) method
Part 2	Determination of silicon by gravimetric method
Part 3	Determination of titanium by the gravimetric method
Part 4	Determination of aluminium
Part 6	Determination of titanium by XRF method

During this revision the method for determination of sulphur by direct combustion method has been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

CHEMICAL ANALYSIS OF FERROTITANIUM

PART 5 DETERMINATION OF SULPHUR BY DIRECT COMBUSTION METHOD

1 SCOPE

This standard (Part 5) describes the method for the determination of total sulphur in the range from 0.005 to 0.2 percent in ferrotitanium.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1472 : 1977	Methods of sampling of ferroalloys for determination of chemical composition (*first revision*)
6226 (Part 2) : 1987	Apparatus for chemical analysis of metals : Part 2 Determination of sulphur by direct combustion

3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1472.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF TOTAL SULPHUR

5.1 Outline of the Method

The sulphur in the sample is converted to sulphur dioxide by combustion in stream of oxygen in a furnace at 1 000°C. It is then absorbed in hydrochloric acid solution and titrated with standard potassium iodate solution, using starch as indicator.

5.2 Reagents

5.2.1 *Dilute Hydrochloric Acid*, 3 percent (*v/v*).

5.2.2 *Starch Solution*

Transfer 1.00 g of pure starch powder and 2 ml of water in a small beaker and stir until a smooth paste is obtained. Pour this mixture into 50 ml of boiling water and cool. Add 1.5 g potassium iodide and stir until dissolved. Dilute to 100 ml. This solution is to be prepared afresh every time.

5.2.3 *Oxygen*, purity 99.5 percent minimum.

5.2.4 *Standard Potassium Iodate Solution* — (1 ml = 0.1 mg S).

Dissolve 0.222 5 g of potassium iodate in 900 ml of water and dilute to 1 litre.

5.2.4.1 *Standard potassium iodate solution* — (1 ml = 0.02 mg S).

Transfer 10 ml of standard potassium iodate solution (5.2.4) to a 500-ml volumetric flask, make up with water and mix well.

NOTE — However, an emperical factor must be determined by analysing ECRM 548-1 (S = 0.030 %) standard sample or any other certified sample.

5.2.5 *Standard Sample*

It should be of known sulphur content in the range of analysis.

5.2.6 *Ceramic Boats/Crucibles*

Preignited at 1 000°C and stored in a dessicator.

5.2.7 *Accelerators*

Iron, tin, etc, free from sulphur.

5.3 Apparatus

5.3.1 The combustion furnace and titration unit as described in IS 6226 (Part 2) is most suitable. However, an induction furnace may also be used with similar purification and titration unit.

5.4 Procedure

5.4.1 Arrange the apparatus as given in IS 6226 (Part 2).

5.4.2 Switch on the furnace with oxygen bubbling through absorption vessel, which is previously filled with dilute hydrochloric acid (3 percent). Add 2 ml of starch solution to the vessel. Adjust the flow of oxygen from 1.0 to 1.5 litre/minute. Fill the burette with potassium iodate solution and add it to the absorption solution dropwise till a blue colour is obtained.

5.4.3 Weigh 1 g of the standard sample and transfer it into a preignited boat/crucible. Add sufficient amount of accelerators (1 g of iron chips and 1 g of tin granules). After the unit has attained required warming that is, about 30 min, introduce the boat/crucible with standard sample and accelerators in the furnace. Close the furnace. Burn the sample while passing oxygen all the time. Titrate continuously with potassium iodate solution at such a rate so as to maintain as close as possible to the original intensity of the blue colour.

NOTES

1 For low sulphur content, the standard potassium solution given in 5.2.4.1 can be used.

2 The end point can be detected if the blue colour is stable for 1 min without further addition of potassium iodate solution. Record the volume in ml of potassium iodate used.

5.4.4 Refill the titration vessel with dilute hydrochloric acid (3 percent). Add 2 ml of starch solution and follow the procedure specified in 5.4.3 using test sample. Titrate the absorption solution with potrassium iodate to the preselected end point and record the volume in ml of potassium iodate used *(A)*.

5.4.5 Determine the blank by placing the same amount of accelerator used in test sample in preignited crucible and following the same procedure as specified in 5.4.3. Deduct the blank value *(B)*, if any from the final calculation.

6 CALCULATION

$$\text{Sulphur, percent by mass} = \frac{(A - B)}{C} \times \frac{F}{10}$$

where

A = volume in ml, of potassium iodate solution required for titration of the sample;

B = volume in ml, of potassium iodate solution required for the titration of blank;

C = mass in g, of standard sample; and

F = factor equivalent to in mg of sulphur for standard solution for potassium iodate.

7 PRECISION

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